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Materials and Theory of topological insulators

ABSTRACT

During the past three years, we have designed and constructed an easily modifiable mini-MBE system that is entirely dedicated to the study of novel materials thin film growth. Using this system, our group has demonstrated the high quality synthesis of three-dimensional topological insulator (TI) thin films the Bi2Te3 family. Bi2Te3 and Bi2Se3 have simple single Dirac cone surface band structures and relatively large bulk band gaps which make them particularly attractive for room temperature device operation [1-2]. Despite the fact that bulk growth techniques for high quality single crystal Bi2Te3 and Bi2Se3 are well known, many modern device applications require thin films.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received	<u>Paper</u>
03/04/2013	2.00 Xiao-Liang Qi, Shou-Cheng Zhang. Topological insulators and superconductors, Reviews of Modern Physics, (10 2011): 0. doi: 10.1103/RevModPhys.83.1057
03/04/2013	3.00 Hai-Jun Zhang, Stanislav Chadov, Lukas Müchler, Binghai Yan, Xiao-Liang Qi, Jürgen Kübler, Shou-Cheng Zhang, Claudia Felser. Topological Insulators in Ternary Compounds with a Honeycomb Lattice, Physical Review Letters, (04 2011): 0. doi: 10.1103/PhysRevLett.106.156402
03/04/2013	4.00 Stanislav Chadov, Xiaoliang Qi, Jürgen Kübler, Gerhard H. Fecher, Claudia Felser, Shou Cheng Zhang. Tunable multifunctional topological insulators in ternary Heusler compounds, Nature Materials, (05 2010): 0. doi: 10.1038/nmat2770
03/04/2013	5.00 X. Zhang, H. Zhang, J. Wang, C. Felser, SC. Zhang. Actinide Topological Insulator Materials with Strong Interaction, Science, (03 2012): 0. doi: 10.1126/science.1216184
03/04/2013	6.00 Binghai Yan, Shou-Cheng Zhang. Topological materials, Reports on Progress in Physics, (09 2012): 0. doi: 10.1088/0034-4885/75/9/096501
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Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

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Scientific Progress

During the past three years, we have worked extensively on the theoretical predictions of topological insulator materials. We discovered three new classes of materials, including the Heusler alloys, the ternary compounds with honeycomb lattice structures and actinide topological insulators. The detailed properties of these materials are described in previous reports and in ARO supported publications listed below. Our theory work has generated great interests worldwide and many experimental groups are working on these materials.

Technology Transfer

MBE Growth of 3D Topological Insulators – Harris MBE Group

During the past three years, we have designed and constructed an easily modifiable mini-MBE system that is entirely dedicated to the study of novel materials thin film growth. Using this system, our group has demonstrated the high quality synthesis of three-dimensional topological insulator (TI) thin films the Bi₂Te₃ family. Bi₂Te₃ and Bi₂Se₃ have simple single Dirac cone surface band structures and relatively large bulk band gaps which make them particularly attractive for room temperature device operation [1-2]. Despite the fact that bulk growth techniques for high quality single crystal Bi₂Te₃ and Bi₂Se₃ are well known, many modern device applications require thin films.

The flexible design of our mini-MBE system has enabled us to accommodate rapid changes necessary for the investigation of new materials. During the latter part of 2011, the Zhang theory group predicted the stoichiometric magnetic topological insulator GdBiTe₃ to be an intrinsic quantum anomalous Hall (QAH) state [3]. The experimental realization of the elusive QAH state is not only of great scientific importance, it is critical to the development of dissipation-less spintronic devices. To date, the thin film growth of this predicted QAH material has not been reported. Any experimental investigation into the exotic behavior of this novel magnetic topological insulator requires the synthesis of high quality GdBiTe₃ thin films. Immediately following the theoretical prediction by Zhang et al., our group began investigating the growth of Gd_xBi_{2-x}Te₃ thin films. We have achieved the synthesis of high quality Gd_xBi_{2-x}Te₃ thin films and demonstrated, for the first time, the possibility of replacing more than 30% of Bi³⁺ with Gd³⁺ in Bi₂Te₃. In what follows, we describe the development of our TI-dedicated mini-MBE system and we report the growth and characterization of high quality single crystalline Bi₂Se₃, Bi₂Te₃, and Gd_xBi_{2-x}Te₃ (0<x<0.6) thin films on c-plane sapphire substrates.

Mini-MBE System Design, Construction and Upgrade

At the start of this project, our efforts were initially concentrated on setting up an old RIBER 32P MBE system previously used by the Yamamoto Group for growth of GaAs quantum well and quantum dot structures for quantum information processing. The RIBER was originally abandoned several years ago due to significant deficiencies within the system (pressure leaks, broken controllers, faulty heaters, etc.). Despite our knowledge of these issues, the resurrection of the RIBER system proved to be more challenging than we originally anticipated due to flood damage and several costly broken and missing parts. After about 6 month's effort, we decided that the time and resources needed to bring up the RIBER system were too great. Our next move was to modify the design of the new mini-MBE system, being assembled for graphene growth, to allow us to deposit topological insulator materials.

Our mini-MBE system for novel materials growth is shown in Fig. 1. This system consists of loading and growth chambers that are connected by a transfer and manipulation system which allows samples to be transferred in and out of growth chamber without breaking vacuum. Substrate heating is provided by a high temperature substrate heating system which is capable of reaching temperatures above 600°C. Our initial design incorporated two cracker cells for Se and Te and one Knudsen-cell for Bi into the growth chamber. Our system was recently modified to include a custom-built high temperature effusion cell capable of operating at temperatures > 1400°C for the sublimation of Gd. Over the past two years, we have experienced very few equipment issues with our mini-MBE system. This has given us the ability to perform almost daily growth runs and to produce many interesting results.

Within the next few months, we will be modifying the mini-MBE again to include a larger growth chamber, which will enable us to incorporate up to 8 different sources, and a precision heating stage, that will allow for finer temperature control. We have also designed an in-situ Hall measurement system, which will enable us to perform transport measurements without exposing our samples to air. We believe these upgrades will significantly expand our abilities to study novel topological systems.

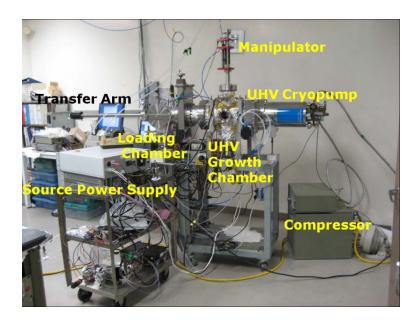


FIG. 1. Custom-built mini-MBE system for novel materials growth.

Growth and Characterization of Bi₂Se₃ thin film

In 2011, we focused on the growth of high quality Bi₂Se₃ thin films using our newly built mini-MBE system. A significant effort was made to optimize growth conditions to achieve high quality single crystal material. We experimented with several substrate growth temperatures, growth rates, flux ratios and substrate preparation methods. The thin film growth of Bi₂Se₃ was found to occur by an island growth mechanism. The surface morphology, crystal quality and band structure measurements for our best samples are summarized below.

The surface morphology of our Bi_2Se_3 thin films is characterized by the atomically flat triangular growth features depicted in the atomic force microscopy (AFM) and scanning electron microscopy (SEM) images shown Fig. 2. The line profile shown in Fig. 2(b), taken across the step edges in Fig. 2 (a), indicates that the triangular terraces are separated by ~ 1 nm, which is consistent with the height of one quintuple layer (QL).

While Bi₂Se₃ grows via van der Waals epitaxial, we found that the quality of the sapphire substrate appears to play a critical role in the alignment of the triangular growth features. Several growths were performed on sapphire substrates from a variety of vendors. With the exception of samples grown on "STEP" sapphire substrates purchased from Shinkosha, the majority of triangle terraces appeared to have a random orientation. Our results suggest that the triangular features found on samples grown on STEP

substrates may have more parallel alignment to neighboring terraces. Figure 3 shows AFM data obtained from Bi₂Se₃ sample grown on conventional and STEP Al₂O₃ substrates.

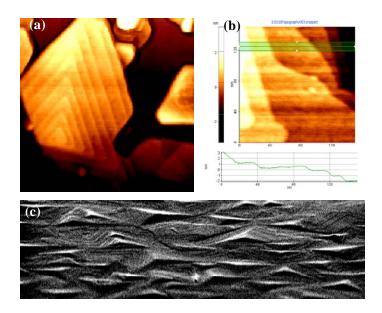


FIG. 2. Surface morphology of Bi₂Se₃ thin films. (a) AFM image of the triangular growth features typically observed on Bi₂Se₃ thin films. (b) Line profile taken across the step edges in image (a). Pre-tilted SEM image of the surface of Bi₂Se₃ thin films.

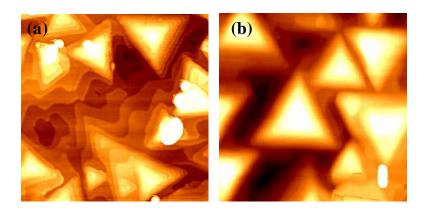


FIG. 3. AFM data from TI films grown on conventional (a) and (b) STEP Al₂O₃ substrates.

X-Ray Diffraction (XRD) and transmission electron microscopy (TEM) studies show that our Bi₂Se₃ thin films consist of nearly perfect single crystals. The XRD rocking curve in Fig. 4 (a) clearly shows the (0 0 3) family peaks of Bi₂Se₃, without any out of plane peaks, indicating that the crystal is aligned in the z-direction. As shown in the high resolution scan of (0 0 6) peak (see inset), the FWHM of the peak is only 0.06°, which suggests a very high crystal quality in the z-direction.

A typical high resolution plane view TEM image [Fig. 4 (b)] reveals that the atomic columns are well-aligned, indicating high quality crystals in those areas. Very clear six fold symmetry can be seen from the TEM selected area diffraction pattern [Fig. 4 (c)], which is believed to be the symbol of defect-free crystals in each domain.

Independent angle-resolved photoelectron emission spectroscopy (ARPES) measurements, performed by Dr. Yulin Chen, confirmed the presence of a topological surface state on our thin films [see Fig. 4 (d)]. The Fermi level for the sample shown in Fig. 4 (d) was found to be located $\sim 0.5 \text{eV}$ above the Dirac point, which indicated that the film was N-type. The unintentional doping of our Bi₂Se₃ thin films has been attributed to surface oxidation and Se vacancies.

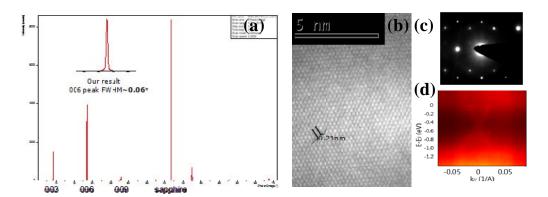


FIG. 4. XRD, TEM and ARPES data. (a) XRD rocking curve showing the (0 0 3) family peaks. Inset of (a) high resolution scan of the (0 0 6) peak. (b) High resolution plane view TEM image of Bi₂Se₃. (c) TEM selected area diffraction pattern. (d) ARPES data confirming the surface state on Bi₂Se₃. Data *courtesy* of Dr. Y-L. Chen.

Growth and Characterization of Bi₂Te₃ thin films

In 2012, we developed a two-step growth method to achieve high quality single crystalline Bi₂Te₃ thin films with wide atomically flat terraces. Without this two-step growth method, the surfaces of our Bi₂Te₃ samples grown at a constant growth temperature were consistently decorated with 3D growth. These 3D growth features were indicative of poor nucleation [see Fig. 5(a)]. To address this issue, we began to first grow a thin nucleation layer at a low growth temperature which was then annealed at a higher temperature under Te overpressure. After the annealing step, the growth was continued a higher temperature. This method has allowed us to achieve good nucleation, eliminate any 3D growth features, and ensure high crystal quality of the films. Figures 5 (b) - (d) show the smooth surface morphology of our Bi₂Te₃ thin films. Our surfaces are characterized by well aligned micron-sized triangular features with 100-300 nm wide atomically flat terraces, separated by step heights of QL. These large domains and atomically flat terraces make our films ideal for experiments using scanning tunneling microscopy or other scanning probe techniques that require large area ultra smooth surfaces.

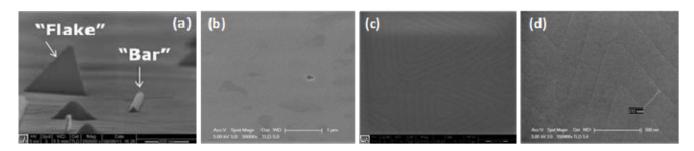


FIG. 5. SEM Data: (a) 3D flake and bar features on constant temperature Bi_2Te_3 growths. (b) Surface of thin nucleation after high temperature annealing. (c) and (d) Smooth surface morphology of thin films grown using the two-step growth recipe.

XRD was used to verify that our films were single crystalline [see Fig. 6(a)]. The cross sectional TEM image shown in Fig. 6(b) indicates high quality layer-by-layer growth. Ex-situ ARPES measurements were performed to confirm the band structure of our Bi₂Te₃ films. The ARPES measurement in Fig. 6(c) shows a Dirac Cone-like band structure indicating the presence of a topological surface state on our Bi₂Te₃ thin films. Issues with surface oxidation and ARPES sample preparation were responsible for the data resolution degradation.

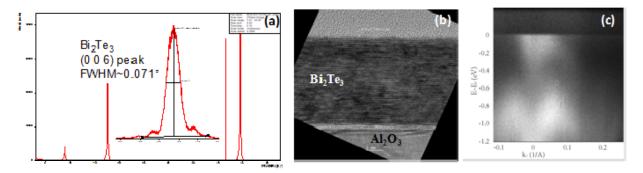


FIG. 6. (a) XRD rocking curve showing the (0 0 3) family of peaks for Bi₂Te₃. Inset of (a) shows an expanded view of the (0 0 6) peak. (b) Cross-sectional TEM image of a 25 nm Bi₂Te₃ sample. Data *courtesy of IBM* Almaden (c) ARPES data showing the band structure of Bi₂Te₃. Data *courtesy of* Dr. Y. L. Chen.

Ex-situ van der Pauw Hall measurements, performed for a variety of growth conditions, indicate that our Bi₂Te₃ films were heavily doped with carrier densities ranging from 10¹⁹ to 10²⁰cm⁻³. Vacancies, anti-site defects, and sample oxidation from exposure to the ambient environment are believed to be responsible for the high doping levels. We also observed a conversion in conduction carrier type. Figure 7 shows a plot of growth temperatures vs. carrier concentration. Samples grown at a lower substrate temperature were found to be N-type whereas samples grown at a higher substrate temperature were P-type. These results suggest that we can tune our growth conditions to achieve a specific carrier type and that intrinsic Bi₂Te₃ films could be achieved by further fine tuning of the growth temperature and by avoiding sample exposure to the air. Similar observations were reported by Wang et al. [4].

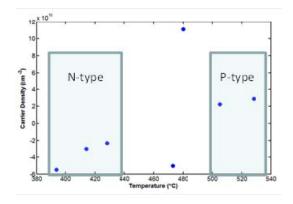


FIG. 7. Carrier concentration vs. growth temperature Hall data obtained using the van der Pauw method at room temperature.

Growth and Characterization of Gd_xBi_{2-x}Te₃ thin films

After optimizing our two-step growth recipe for high quality ultra smooth Bi_2Te_3 thin films, our efforts have been focused on Gd-doping of Bi_2Te_3 . Our initial experiments were carried out by incorporating a small percentage of Gd (0 < x < 0.6)) into Bi_2Te_3 in order to study gradual changes in the material properties as we move toward our goal of Bi:Gd = 1:1 (x = 1). All $Gd_xBi_{2-x}Te_3$ (0<x<0.6) growths were performed using our two-step growth recipe at a rate of ~ 1 nm/min.

Similar to our Bi_2Te_3 growth, the surface morphology of our $Gd_xBi_{2-x}Te_3$ thin films is characterized by well aligned 0.5 µm-sized triangular features with ~ 30 -100 nm wide terraces [see Figs. 8(a) and 8(b)]. Unlike our Bi_2Te_3 films, small dot-like protrusions are consistently observed on Gd-doped films [see Figs. 8(c) and 8(d)]. The density of dots was found to increase with higher growth temperature, which suggests that this effect is related to surface phase segregation. Cleaving of the top surface of our $Gd_xBi_{2-x}Te_3$ thin films was found to completely eliminate the dot features.

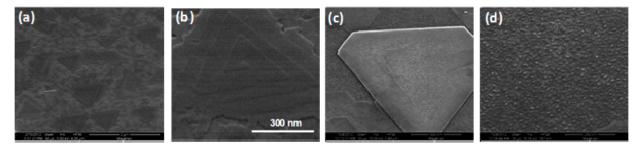


FIG. 8. SEM Data: (a) Typical surface morphology of $Gd_xBi_{2-x}Te_3$ thin films. (b) Triangular feature composed of terraces separated by ~ 1 nm high steps. (c) Extreme example of a phase segregated surface on a $Gd_xBi_{2-x}Te_3$ sample. (d) Higher resolution image of the small dot features shown in (c).

XRD was used to determine the crystal quality of our thin films and to confirm the possibility of growing GdBiTe₃ in the Bi₂Te₃ crystal structure. Figures 9(a) and 9(b) show the (0 0 6) peaks of the XRD rocking curves and the lattice constant variations for various Gd concentrations, respectively. The in-plane lattice constant was found to increase with increasing Gd concentration whereas the out-of- plane lattice was found to decrease with increasing Gd concentration.

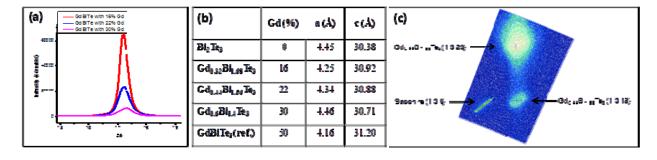


FIG. 9. (a) XRD rocking curves of (0 0 6) peak for various Gd concentrations. (b) In- and out-of-plane lattice constants for various Gd concentrations. (c) 2D reciprocal space mapping of Gd_{0.44}Bi_{1.56}Te₃ thin film.

ARPES and magnetic measurements were performed on our $Gd_xBi_{2-x}Te_3$ thin films to look for the QAH signatures in 3D TI materials: a gap opening in surface state and ferromagnetic ordering. Figure 10 shows the ARPES measurements obtained from a P-type $Gd_{0.32}Bi_{1.68}Te_3$ sample that was cleaved inside of the ARPES measurement system to remove the top surface layers. Potassium doping, by surface sputtering, was used to raise the Fermi level so that the surface state was visible. Figure 10 shows that a surface state is clearly present in our $Gd_{0.32}Bi_{1.68}Te_3$ thin film. These measurements provide definitive confirmation that $Gd_xBi_{2-x}Te_3$ is another new topological insulator and could possibly be used as a platform to realize QAH. However, careful analysis of the data suggests that a gap in the surface state was not observed for $Gd_xBi_{2-x}Te_3$ samples with 0 < x < 0.6.

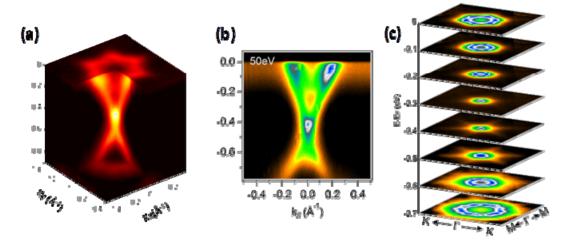


FIG. 10. ARPES data for a $Gd_{0.32}Bi_{1.68}Te_3$ sample. (a) Three-dimensional illustration of bandstructure after K-doping. (b) Bandstructure along the K- Γ -K direction using a photon energy of 50 eV. (c) Constant energy contours of the bandstructure. Data *courtesy* of Dr. B. Zhou.

Magnetic measurements performed to determine the magnetic properties of our films are shown in Fig. 11. X-ray Magnetic Circular Dichroism (XMCD) hysteresis measurements at normal and 20° grazing angle incidence indicate a paramagnetic response at temperatures above 1.5K [see Fig.11(a) inset]. The magnetic response as a function of temperature is shown in Fig.11(a). This plot suggests a possible phase transition to the anti-ferromagnetic state at 1.5K. The paramagnetic response of our $Gd_xBi_{2-x}Te_3$ thin films indicates that Gd clusters were not formed during growth. SQUID magnetic hysteresis measurements obtained on a $Gd_xBi_{2-x}Te_3$ sample for $T \ge 10K$ are shown in Fig. 11(b).

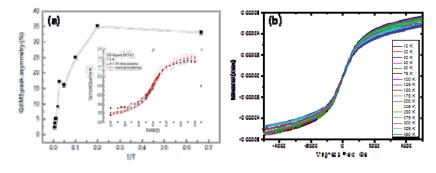


FIG. 11. Magnetic measurements performed on a Gd_{0.6}Bi_{1.4}Te₃ sample. (a) XMCD Data: Gd M5 peak asymmetry vs. temperature showing a possible phase transition at 1.5K. Inset of (a) Paramagnetic hysteresis response of Gd for both normal and 20° grazing angle incidence at 1.5 K. Data *courtesy* of Dr. T. Hesjedal et al. in Diamond Light Source at UK. (b) SQUID magnetic hysteresis measurement showing paramagnetic response at T≥10K. Data *courtesy* of IBM Almaden.

Conclusions

High quality, single crystal Bi_2Se_3 and Bi_2Te_3 thin films were grown on c-plane sapphire substrates using our mini-MBE system. While the growth of our Bi_2Se_3 films utilized a constant temperature growth recipe, our highest quality Bi_2Te_3 films were achieved using a unique two-step growth method. The surface morphologies of our optimized Bi_2Te_3 and Bi_2Se_3 thin films were characterized by large triangular features with wide atomically flat terraces, separated by step heights of ~ 1 QL. XRD and TEM measurements indicated that single crystal film growth was achieved. ARPES measurements on Bi_2Se_3 and Bi_2Te_3 thin films verified the presence of topological surface states.

We also demonstrated the possibility of replacing more than 30% of Bi^{3+} with Gd^{3+} in Bi_2Te_3 . Using the two-step growth method, we have achieved high quality $Gd_xBi_{2-x}Te_3$ thin films. Although a paramagnetic response is observed in $Gd_xBi_{2-x}Te_3$ (0<x<0.6) thin films above 1.5K, it may still be possible to realize ferromagnetism or anti-ferromagnetism in $Gd_xBi_{2-x}Te_3$ thin films by incorporating more Gd or other magnetic dopants [5]. ARPES measurements confirmed the presence of a non-trivial topological surface state in our $Gd_xBi_{2-x}Te_3$ thin films, which suggests that we are one step closer to realizing a QAH state.

We plan to continue to study the growth mechanisms, material properties and transport of 3D topological insulator thin films. We are currently investigating the possibility of including other magnetic dopants into our $Gd_xBi_{2-x}Te_3$ thin films to enhance magnetic order and realize QAH.

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Theoretical predictions of topological insulator materials – Zhang Theory Group

During the past three years, we have worked extensively on the theoretical predictions of topological insulator materials. We discovered three new classes of materials, including the Heusler alloys, the ternary compounds with honeycomb lattice structures and actinide topological insulators. The detailed properties of these materials are described in previous reports and in ARO supported publications listed below. Our theory work has generated great interests worldwide and many experimental groups are working on these materials.

We have worked extensively with Harris group on the growth of topological insulator materials in general, and on the magnetic topological insulators GdBiTe in particular. The goal is to search for the quantized anomalous Hall effect in this magnetic topological insulator.

With the ARO support, we completed two commissioned review articles on topological insulators. The general review on "Topological insulators and superconductors" was published in the Review of Modern Physics. It summarizes the model and materials for topological insulators, and discusses various physical effects of these materials. The materials specific review on "Topological materials" was published in the Report of Progress of Physics. It summarizes the status of all topological materials predicted and discovered to date, and presents the overall systematic trends, which could guide our future search.

Publications supported by ARO funding W911NF-09-1-0508

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Awards

Europhysics Prize, Awarded by the European Physical Society. (2010)

Election to the American Academy of Arts and Sciences. (2011)

Oliver Buckley Prize, Awarded by the American Physical Society. (2012)

Dirac Medal and Prize, Awarded by the International Center for Theoretical Physics. (2012)